N64-27900
(ACCESSION NUMBER) 47 (PAGES) CR-58204 (NASA CR OR TMX OR AD NUMBER)
(THRU) 1 (CODE) 17 (CATEGORY)
FACILITY FORM 602

# RESEARCH ON BEARING LUBRICANTS FOR USE IN HIGH VACUUM

47 p.

ANNUAL SUMMARY REPORT
23 March 1962 - 22 April 1963

Contract No. NAS8-1540
Control No. TP 85-137

M.R.I. Project No. 2492-E

OTS PRICE

XEROX $ 4.60 ph

MICROFILM $ ______

For

National Aeronautics and Space Administration
Marshall Space Flight Center
Huntsville, Alabama
Attn: Office of Procurement and Contracts, M-P&C-CA

MIDWEST RESEARCH INSTITUTE

# RESEARCH ON BEARING LUBRICANTS FOR USE IN HIGH VACUUM

by

Vern Hopkins
D. H. Gaddis

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## PREFACE

This report describes the activities conducted between 23 March 1962 and 22 April 1963, on the subject project, Contract No. NAS8-1540, Control No. TP 85-137, MRI Project No. 2492-E. This program is administered by the Mechanical Engineering Branch, Propulsion and Vehicle Engineering Division of the George C. Marshall Space Flight Center. Mr. Eugene C. McKannan is the technical representative of the NASA contracting officer.

The work on this program is under the technical supervision of Mr. Vern Hopkins, Head of the Materials Section. The Project Leader is Donald H. Gaddis. The authors acknowledge the valuable contributions of the following MRI staff members:

Engineering Division
- Ronald D. Hubbell - lubricant-binder evaluations
- Frank J. Barker - instrumentation design
- B. Kinder and R. Schaefer - laboratory work

Chemistry Division
- Donald W. Rathburn - binder material formulation

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30 August 1963

## ABSTRACT

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Binder development studies have been performed to develop better binders for solid lubricant films during the past year. Potassium silicate was selected as a basic binder. Preliminary results show that the wear-life of this binder material may be increased by additives such as sodium phosphate, potassium phosphate, sodium borate, or sodium fluoride.

A search for additional lubricants or lubricant film components was conducted. Nine potential materials were selected for formulation and evaluation.

A gear apparatus and pellet apparatus were designed, built, and used to investigate solid lubricant film wear-life in air at room temperature. Wear characteristics of MLF-5 ($MoS_2$ + graphite + gold/sodium silicate) obtained in the early runs with the pellet apparatus are presented graphically.

MLF-5 and other solid lubricant films were applied to a number of parts and components for Marshall Space Flight Center and other concerns.

A number of modifications which were made on the vacuum friction apparatus to improve its over-all operating efficiency are described.

Author

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## SUMMARY

Since the binder in an inorganic solid lubricant film is as important as the lubricant, a great deal of effort has been spent on the development and evaluation of inorganic binders. Potassium silicate (Kasil 88) was selected as a base material. Kasil 88 appears to adhere better to 440C stainless steel than sodium silicate (k) and it is also more resistant to degradation from water. Binders were formulated by adding to the Kasil 88 sodium phosphate, potassium phosphate, sodium borate or sodium fluoride one at a time in varying amounts.

Several methods of applying binders and curing binders onto 440C substrate metal were investigated. One method which consisted of spraying the binder onto a hot substrate (300°F), appeared to be as good as the conventional long-cure method.

Three ways of evaluating binder performance were investigated. The most consistent results were obtained by determining wear-life in a nitrogen atmosphere. The wear-life method used was to slide three pellets over a wear plate while they were under a light load (50 gm/contact). An outstanding binder has not yet been formulated; however, binder wear-life can be improved by the addition of alkali and alkaline earth metals.

A lubricant film was formulated by substituting Kasil 88 with 9.4 per cent $Na_3PO_4$ for sodium silicate in the MLF-5 formulation. The frictional behavior of this modified MLF-5, solid lubricant film, was found to be essentially the same as MLF-5.

One hundred sixty-five materials were screened on the basis of their physical and chemical properties in a search for potential lubricating materials. Twenty-two of these materials were selected for further screening under sliding friction. Nine of these materials $BiI_3$, $Bi_2Te_3$, $CdBr_2 \cdot 4H_2O$, $AuTe_2$, $MoSe_2$, $AgBrO_3$, $AgCl$, $WS_2$ and $WSe_2$ exhibited friction coefficients of 0.22 or less and are considered good enough to warrant incorporation into a solid lubricant film for further screening.

Two testers were designed and fabricated to investigate lubricant wear-life on pellets and gears under light loads, at room temperature and in an air atmosphere. Data from early runs indicate that the longest wear-life is obtained from a bonded inorganic solid lubricant film when it is mated with a smooth surface.

# I. INTRODUCTION

A program is being conducted for the NASA Marshall Space Flight Center to develop bonded, inorganic, solid lubricant film suitable for use in air and outer space environments. In this program, solid lubricant films are being investigated at ambient pressures from normal atmospheric to $10^{-7}$ Torr and temperatures from 80° to 400°F while under light loads (in the range of 2 to 14 psi).

During the first year of the program[1] an apparatus was designed and built to investigate the frictional behavior of solid lubricant films over the required range of environmental conditions. A great number of materials were considered for use as potential lubricants or lubricant film components. Included among these materials were noble metals, low melting point metals, precious stones, molybdenum disulfide and other inorganic compounds. A large number of these materials were selected, formulated into composite films, and investigated along with many known solid lubricant films. Frictional and wear characteristics of the films investigated were compared with those of a film of electroplated gold. This comparison, in turn, made possible the judging of the relative performance of the films. Five of the solid lubricant films investigated exhibited better performance than electroplated gold film. One of these films was selected by NASA for use in particular applications and was designated MLF-5.*

# II. BINDER DEVELOPMENT STUDIES

## A. Requirements for Solid Lubricant Binders

In the formulation of a bonded-type inorganic solid lubricant film, the binder is as important as the lubricant. The function of a binder is to bind a lubricant to a surface to be lubricated. A binder usually forms a matrix type structure and as such holds the lubricant in many tiny reservoirs. If the binder is easily fractured or abraded then the composite lubricant film wears rapidly and the full benefit of the lubricant is not realized.

During this report period (March 1962 - April 1963), much effort has been devoted to the development and evaluation of improved binder materials. The over-all objectives of this effort are to develop binders that are (1) wear resistant, (2) compatible with selected lubricants, (3) highly resistant to damage from water, rocket fuels, oxidizers, and the earth's atmosphere,

* MLF-5 solid lubricant film is described in detail in Appendix C.

(4) capable of bonding tightly to the substrate surface, and (5) able to cure (bond) at a moderate temperature. When binder curing temperatures are too high, the mechanical properties of the substrate (base metal) are significantly reduced or impaired.

The lubricant-binder systems being developed are required to withstand the environment of 70° to 400°F in an air atmosphere and at ambient pressures to $10^{-6}$ Torr or less. They must also remain stable over the range of temperature and humidity existing in the earth's atmosphere. To meet these requirements, glazes properly modified to the specified conditions and over-all objectives have been formulated. The problem is somewhat similar to preparing porcelain enamel to metals, or glazes to ceramic surfaces. However, the binder material for lubricants must cure at temperatures much lower than required for such enamels or glazes.

## B. Selection of Materials and Binder Formulations

Present binder materials under consideration are based on the amorphous or glassy character of certain silicates, borates and phosphates. The amorphous nature of these materials results from the formation of long polymeric chains of the central atoms with oxygen (-O-Si-O-Si-O-, -O-B-O-B-O-). This structure gives good adhesion to substrate materials while forming thin continuous films. Incorporation of lubricant materials into the binder yields a film in which the binder acts as a matrix for holding the lubricant particles.

In a review of the literature on solid lubricant binder films, the use of sodium silicate was frequently encountered. Very little mention of potassium silicate was found, although both potassium silicates and sodium silicates have special characteristics which make them suitable as solid lubricant binder materials. When exposed to moist air, a sodium silicate tends to "frost" or effloresce by the formation of certain sodium carbonate hydrates. Dried films of potassium silicate have much less tendency to effloresce on exposure to moist air.

The addition of alkali earth metal or alkaline earth metal to a potassium silicate has improved its adherence to glass substrates, and may also improve its adherence to a metal substrate as well as increase the flexibility of a cured film. An increase in binder flexibility reduces the tendency of a binder to crack when it is stressed. Either improvement is expected to enhance the properties of a potassium silicate as a solid lubricant binder.

Initially three potassium silicates were investigated for use as the basic binder material. These three potassium silicates have the following $K_2O:SiO_2$ ratios: Kasil No. 1, 1:2.50; Kasil No. 88, 1:2.20; and Kasil No. 6, 1:2.10 (each has a different viscosity). Kasil No. 88 was selected as the most suitable potassium silicate to modify with various selected additives (alkali and alkaline earth metals).

Binders were formulated by adding sodium phosphate, potassium phosphate, sodium borate, and sodium fluoride individually in varying amounts to Kasil No. 88. The silicate solutions were then diluted approximately 3 to 1 with water, forming a solution of 10 to 15 per cent solids. Next, they were applied to 440C stainless steel substrate metal and cured. After curing, the modified silicates were ready for evaluation.

## C. Evaluation of Formulated Binders

Several evaluation techniques were considered and tried in an effort to find an effective method of evaluating formulated binders. The method found most effective consists of applying a binder to three pellets rigidly mounted in a pellet holder and rubbing them against a wear track plate on the vacuum friction apparatus in a nitrogen atmosphere. Binder performance is judged on the basis of frictional torque data and wear-life data. Film failure (i.e., the instant the binder wears through to the substrate) is detected by an electrical-contact resistance-measuring device described in Appendix A.

The first frictional torque and wear-life data obtained from binder screening runs (see Table B-I in Appendix B) exhibited considerable scatter. All of these runs were characterized by considerable pellet holder vibration and chatter as well as by widely fluctuating frictional torque.

Two wear-track hardnesses, Rockwell C 15 to 20 and 55 to 59, were used during the initial binder wear-life studies in air. It can be seen in Table B-I, that in all cases except one, shorter wear-lives were experienced with softer wear tracks.

In attempting to account for the scatter, consideration was given to the chemical stability and concentrations of the binder solutions, binder application procedures, cure cycles, wear-track and pellet preparation procedures, film failure detection methods, and evaluation conditions such as load, speed, temperature, and environmental atmosphere.

At the end of each of these early binder screening runs, the pellets and wear-track plates were examined. The contact surfaces of the pellets and wear tracks contained irregular smears of a black granular material. They also contained, in lesser quantities, traces of a reddish brown material. In addition, many discrete particles of each material were found distributed over the entire surface of the wear-track plate. Since the wear-track plates and pellets were made of 440C stainless steel (high carbon, martensitic steel), the foreign material was believed to be iron oxide (s). Because this oxide (s) is highly abrasive and its presence would tend to confuse interpretation of the data, additional binder screening runs were performed in an inert (nitrogen) atmosphere. The results of these runs show that the scatter is much reduced (see Tables B-II and B-III in Appendix B).

Several techniques of applying binders to the ends of the 440C pellets were investigated. The results of this investigation are presented in Table B-II in Appendix B. The application techniques investigated were:

<u>Method A</u>: The binder solution was sprayed onto the pellets with an air brush at such a rate that excessive moisture would not collect on the pellets. Drying of the binder coating was aided by heating the pellet surface to approximately 140°F with an infrared heat lamp during the spraying process. The binder coating was cured 1 hr. at 140°F and then 16 hr. at 200°F in an air oven.

<u>Method B</u>: The binder material solution was sprayed onto the pellets in the same general manner as in Method A, except that spraying was stopped at regular intervals to allow more complete drying. The final coating was built up in six steps (or layers) allowing 45 min. drying between each spray application. The curing cycle was the same as in Method A.

<u>Method C</u>: The binder material was applied by placing one drop of concentrated solution on the end of each pellet. The pellets were at room temperature (75°F). The curing cycle consisted of room temperature air-drying for 4 hr. followed by 16 hr. at 200°F in an air oven.

<u>Method D</u>: The binder material solution was applied to the pellets in the same manner as in Method A except that the pellet temperature was maintained at 215°F during application. The binder was cured for 16 hr. at 215°F in an air oven.

<u>Method E</u>: This method was identical to Method D except that the pellet temperature was maintained at 300°F during binder application. The binder was cured for 1/2 hr. at 300°F in air.

Method F: This method was identical to Method E except that the pellet temperature was maintained at 395°F during application and the binder was cured for 1/2 hr. at 395°F in air.

Data in Table B-II show that it is possible to cure potassium-silicate type binders rapidly by spraying them onto a hot substrate. When the hot substrate short-cure technique is used, binder wear-life is comparable to that of a binder cured with the conventional technique (1 hr. at 140°F and 16 hr. at 200°F in an air oven). The purpose of curing is to drive off the moisture in the film. In the conventional curing method, part of the moisture is driven from deep in the film through the cured or partially cured film. Attempts to hasten the curing process by using higher temperatures promotes the formation of blisters in the film. When the film is sprayed onto a hot substrate, the minute spray particles cure very rapidly. Most of the moisture has a very short distance to travel to escape from the film.

The results of this binder work suggest that it may also be possible to employ such a technique to cure rapidly a formulated solid lubricant film. The use of a rapid cure technique would save both time and money in lubricating machine elements with potassium-silicate-bonded solid-lubricant films.

The procedures used in the early binder studies were modified after an investigation of possible causes of scatter in the binder wear-life data. The following procedures were found to be the most satisfactory.

1. Binder material solutions containing additives were used within 2 days after binder formulation.

2. Binders were applied to the pellets maintained at a temperature of 300°F.

3. The binder-coated pellets were cured in an air oven for at least 12 hr. at 180° to 190°F.

4. Binder-screening runs were performed within a 24-hr. period following curing.

These binder-screening runs were performed under the following test conditions:

Load - 50 gm/contact (2.2 psi projected area)
Speed - 900 rpm (765 fpm rubbing speed)
Environmental atmosphere - nitrogen
Wear-track material - 440C stainless steel
Wear-track hardness - 55 to 59 Rockwell C
Wear-track roughness - 6 to 7 rms
Wear-track temperature - 70° to 90°F
Film failure criterion - first instant of metal-to-metal contact

The data collected from the runs performed under these revised procedures and conditions are presented in Table B-III in Appendix B.

A comparison of the results for binder-screening runs in a nitrogen atmosphere to the results for runs in air show that:

1. Longer wear-lives are obtained in a nitrogen atmosphere.

2. Data scatter is generally less for runs made in the nitrogen atmosphere.

## D. Improved Binder Material in Composite Film

Sample No. 7 (potassium silicate + 9.4 per cent $Na_3PO_4$) was selected as an improved binder material to be investigated in an established lubricant film formulation. This binder was used in place of sodium silicate in the MLF-5 formulation. The resulting film was called Mod. MLF-5, and was subjected to frictional behavior runs to determine the frictional characteristics.

The results of the frictional behavior runs are presented in Table I. There is no significant difference between friction coefficients for Mod. MLF-5 and MLF-5 in air and vacuum atmospheres at each test temperature.

## E. Other Methods of Evaluating Binders

Several methods of evaluating the developed binder materials were considered during the initial phase of the binder development studies. In one method, the adhesion of the binder material to 440C stainless steel was determined.

TABLE I

FRICTION RESULTS FROM FRICTIONAL BEHAVIOR RUNS

Load: 50 gm/contact (2.2 psi)
Wear-track roughness and hardness:
4 - 6 rms and 55 - 59 Rockwell C
Run Duration: 60 min.
Speed: 900 rpm (765 fpm)

| Lubricant | Wear Track Temperature (°F) | Environmental Pressure (mm. Hg) | Friction Coefficient Range |
|---|---|---|---|
| MLF-5[a] | 80 | 760 | 0.12-0.21 |
| MLF-5 | 250 | 760 | 0.05-0.07 |
| MLF-5 | 400 | 760 | 0.10-0.15 |
| MLF-5 | 80 | $10^{-7}$ | 0.07-0.10 |
| MLF-5 | 250 | $10^{-7}$ | 0.09-0.10 |
| MLF-5 | 400 | $10^{-7}$ | 0.04-0.14 |
| Mod. MLF-5[b] | 80 | 760 | 0.17-0.22 |
| Mod. MLF-5 | 250 | 760 | 0.05-0.07 |
| Mod. MLF-5 | 400 | 760 | 0.06-0.11 |
| Mod. MLF-5 | 80 | $10^{-7}$ | 0.09-0.12 |
| Mod. MLF-5 | 250 | $10^{-7}$ | 0.08-0.11 |
| Mod. MLF-5 | 400 | $10^{-7}$ | 0.08-0.11 |

a/ MLF-5 solid lubricant film developed by MRI, see Appendix C for description.

b/ Mod. MLF-5 solid lubricant film same as MLF-5 except potassium silicate containing 9.4 per cent of $Na_3PO_4$ was used as the binder instead of sodium silicate.

One end of two pellets was coated with the binder and then one was positioned on top of the other with the coated ends in contact. The pellets were then placed in an oven to cure the binder. After curing, the bending moment required to break them apart was determined. The maximum bending moment transmitted by the binder from one pellet to the other was a measure of binder adherence to the substrate metal or to itself.

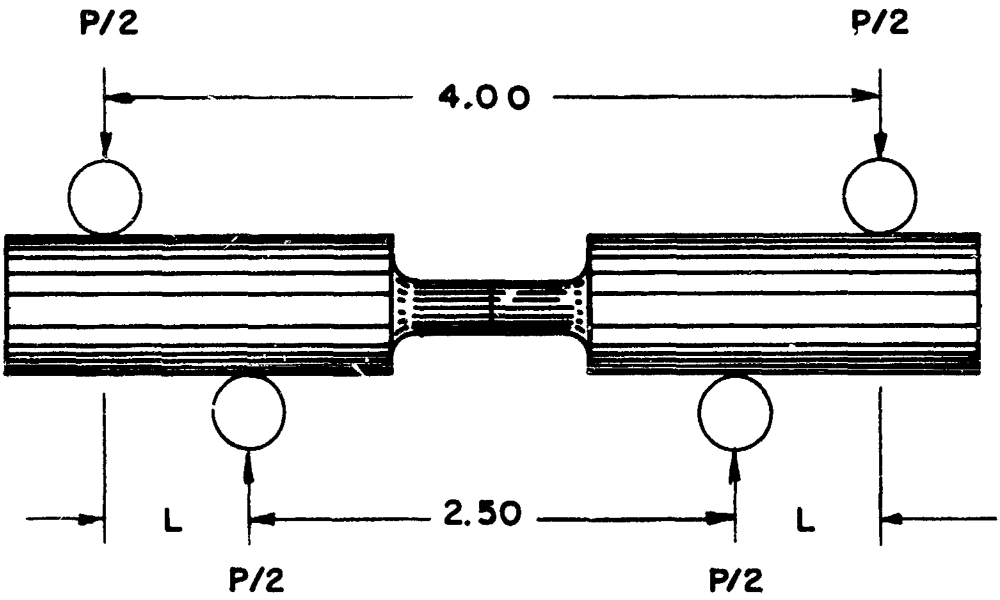
A two-point loading method was employed which produced a constant moment along the 2.5-in. gage length. The bonded joint was located near the center of this gage length (see Fig. 1). The results of this method of evaluating binders are shown on Table II. The data presented show a large amount of scatter. It is believed the scatter was caused by incomplete and uneven bonding of one pellet to the other.

Several procedures of applying the binder to the pellets and subsequent joining together were tried. The first procedure consisted of dipping the end of each pellet in a highly concentrated solution of the binder material, bringing the coated ends together, and placing the pair, one on top of the other, in a holding fixture to cure. The binder was then cured in an air oven.

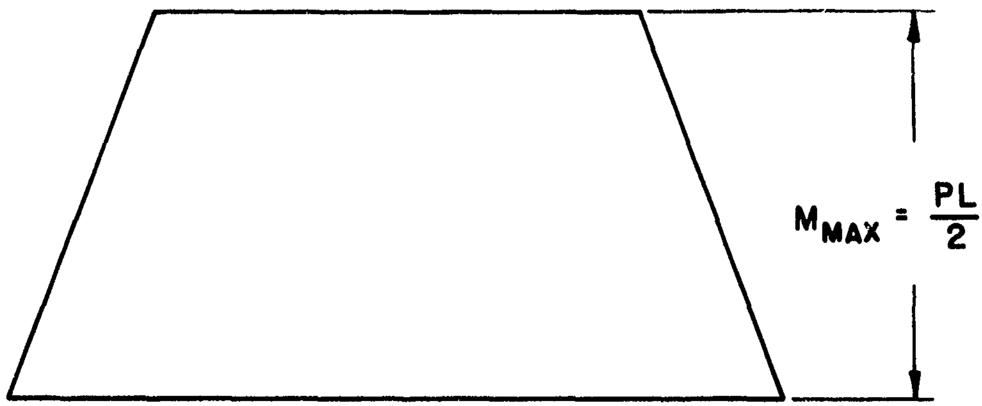
A second procedure consisted of applying several thin coats of binder material with an air brush to the ends of the pellets until a thickness of 0.005 in. had been built up. After each application with the air brush, the coating was allowed to dry slightly. The pellets were then dipped in the concentrated binder solution and joined together for curing as in the first procedure.

A third procedure consisted of applying several thin coats of binder material with an air brush as before. However, before joining the pellets together, one heavy spray coat was applied. The pellets were then placed one on top of the other before the coating dried and cured as before.

When the pellets were broken apart, voids or pockets in the coating on the contact surface of each pellet could usually be seen regardless of the method used to apply the binder. The outline or shape of each void on one pellet matched that on the other. The voids appeared very similar to gas pockets such as are found in imperfect castings. The depth of the voids appeared to be approximately equal to the thickness of the last coating of binder material applied to the pellets. When the pellets were joined together using the first procedure, the bottom of the void areas exposed uncoated substrate metal. When the pellets were spray coated prior to joining together as in the second or third method, a coating of binder material was observed on the bottom of the void areas. The presence of voids indicated excessive shrinkage of binder solutions when they are used to bond the pellets together.




TWO-POINT LOADING METHOD




MOMENT DIAGRAM

Fig. 1 - Two-Point Loading Method

TABLE II

RESULTS OF BINDER ADHERENCE INVESTIGATION

| Binder | Maximum Load at Rupture (lb.) | Maximum Tensile Stress (psi) |
|---|---|---|
| Sodium Silicate | 57.9 | 12,500 |
| Sodium Silicate | 70.0 | 15,150 |
| Sodium Silicate | 7.0 | 1,530 |
| Potassium Silicate No. 6 | 15.6 | 3,390 |
| Potassium Silicate No. 6 | 13.7 | 2,960 |
| Potassium Silicate No. 6 | 18.7 | 4,060 |
| Potassium Silicate No. 88 | 13.4 | 2,920 |
| Potassium Silicate No. 88 | 11.5 | 2,510 |
| Potassium Silicate No. 1 | 22.9 | 4,980 |
| Potassium Silicate No. 1 | 11.4 | 2,480 |
| Potassium Silicate No. 1 | 8.6 | 1,860 |
| Potassium Silicate Std. | 12.1 | 2,630 |
| Potassium Silicate Std. | 14.0 | 3,040 |
| Potassium Silicate Mod. No. 2 | 8.4 | 1,825 |
| Potassium Silicate Mod. No. 2 | 5.7 | 1,242 |

The fewer the number of voids or pockets in the surface of the coating, the greater the bending moment required to break the pellets apart. In all cases the binders appeared to form a good bond to the substrate metal. After the pellets had been broken apart, the remaining coating was given a simple scratch test with a small needle. The potassium silicate binders adhered better to the substrate than did sodium silicate. That is, it was more difficult to scratch or scrape the potassium silicate coatings off the substrate. Cured films of potassium silicate binders, both straight and modified, appear to be more resistant to the moisture than sodium silicate. It was possible to remove a cured sodium silicate film by rubbing lightly after soaking it in detergent and water. Cured films of potassium silicate, straight and modified, could not be removed in this manner. It was necessary to abrade these binders off the 440C substrate on a lapping plate or with a vapor hone.

The results of this binder evaluation method indicated that good bonds were achieved between the substrate and the binder material, and that potassium silicate adhered better then sodium silicate. The large amount of scatter in the data suggested that the method of bonding the two pellets together was inadequate. None of the procedures of bonding the pellets together which were tried were found effective.

A third method of evaluating developed binders consisted of applying a thin coating of the binder to a 440C stainless steel bar, 1/4 in. thick, 1 in. wide, and 12 in. long. After the film had been cured, one end of the bar was rigidly held while the other end was deflected a fixed amount. The end deflection of the cantilevered bar produced a varying strain in the binder coating along the length of the bar. Fracture of the binder across the width of the bar occurred along the bar from the fixed end until a point was reached where the strain was such that the binder would not fracture. The measurement of maximum strain at this point provided a means of obtaining the maximum tensile strength of the binder material. However, the data were so widely scattered that this method of evaluating binders was also abandoned.

## III. SEARCH FOR POTENTIAL LUBRICANT MATERIALS

In the development of inorganic solid lubricant films a great variety of materials are under consideration for use as lubricant pigments or lubricant film components. Included among these materials are noble metals, low-melting-point metals, precious stones, molybdenum disulfide, graphite and many other inorganic compounds.

During the first year of this research program many known solid lubricant films were selected and investigated. In addition, some 300 inorganic materials (having melting points between 500° and 1000°F) were sorted from an IBM deck of 22,000 materials compiled on an earlier Air Force program.[2] This group of materials was screened by eliminating those which:

- Oxidize below 400°F in air;
- Are reactive with water;
- Have high vapor pressures (above 1/2 atmosphere) at 400°F;
- Are hygroscopic; or
- Are soluble in water.

During this past year's work the original group of 300 inorganic materials was re-examined for potential lubricant materials. Materials were selected by eliminating only those which:

Oxidize below 400°F in air;
Are reactive with water; and
Have high vapor pressures at 400°F.

This screening differs from the initial screening by the deletion of the hygroscopic and water insolubility requirements. This deletion was made in the screening because it is believed that a binder material which cures to a water insoluble state may provide adequate protection for the lubricant. If required, additional protection could be provided by applying a very thin coating of "Teflon" TFE or FEP fluorocarbon resin.

One hundred sixty-five materials were obtained in this screening. These materials have been further screened by selecting those which have crystal structures similar to other known solid lubricants such as $MoS_2$.

Other potential lubricant materials selected include $MoSe_2$, $MoTe_2$, CdTe, $WSe_2$ and $WS_2$. These lubricant materials have been investigated on a high-temperature, solid-film lubricant project for the Air Force.[3,4]

Twenty-two selected potential lubricant materials were next subjected to preliminary screening runs in order to obtain a measure of their lubricity. Preliminary screening runs consist of lightly burnishing a potential lubricant material onto the flat ends of three vapor honed pellets which are rigidly held in a pellet holder. The minimum coefficient of friction is recorded as the pellets are rubbed against a wear-track plate on the vacuum friction apparatus The results of the preliminary screening runs are presented in Table III. The following nine potential lubricant materials were selected from this group for futher investigation.

| | |
|---|---|
| Bismuth iodide | $BiI_3$ |
| Bismuth telluride | $Bi_2Te_3$ |
| Cadmium bromide | $CdBr_2 \cdot 4H_2O$ |
| Gold telluride | $AuTe_2$ |
| Molybdenum diselenide | $MoSe_2$ |
| Silver bromate | $AgBrO_3$ |
| Silver chloride | AgCl |
| Tungsten disulfide | $WS_2$ |
| Tungsten diselenide | $WSe_2$ |

Preliminary screening runs are performed under the following conditions:

| | |
|---|---|
| Environment | Air atmosphere |
| Load | 50 gm/contact (2.2 psi projected area) |
| Speed | 900 rpm (765 fpm rubbing speed) |
| Wear-track temperature | 80° - 90°F |
| Wear-track roughness | 4 - 6 rms |
| Wear-track hardness | 55 - 59 Rockwell C |

TABLE III

FRICTION RESULTS FROM PRELIMINARY SCREENING RUNS

Load: 50 gm/contact (2.2 psi)

Speed: 900 rpm (765 fpm)
Atmosphere: Air

Wear-Track Temperature, Roughness, and Hardness: 80°F, 4 to 6 rms, and 55 to 59 Rockwell C

| Lubricant Material | Coefficient of Friction (min.) |
|---|---|
| Antimony sulfide, $Sb_2S_3$ | 0.34 |
| Arsenic sulfide, $As_2S_2$ | 0.33 |
| Bismuth iodide, $BiI_3$ | 0.16 |
| Bismuth sulfide, $BiS_3$ | 0.50 |
| Bismuth telluride, $Bi_2Te_3$ | 0.12 |
| Cadmium bromide, $CdBr_2 \cdot 4H_2O$ | 0.13 |
| Cadmium iodide, $CdI_2$ | 0.55 |
| Cadmium hydroxide, $Cd(OH)_2$ | 0.30 |
| Cadmium telluride, CdTe | 0.30 |
| Gold telluride, $AuTe_2$ | 0.18 |
| Lead iodide, $PbI_2$ | 0.29 |
| Lead sulfide, PbS | 0.44 |
| Molybdenum carbide, $Mo_2C$ | 0.55 |
| Molybdenum diselenide, $MoSe_2$ | 0.09 |
| Molybdenum ditelluride, $MoTe_2$ | 0.28 |
| Platinum dioxide, $PtO_2$ | 0.38 |
| Silver bromate, $AgBrO_3$ | 0.18 |
| Silver chloride, AgCl | 0.18 |
| Silver iodide, AgI | 0.32 |
| Tungsten diselenide, $WSe_2$ | 0.22 |
| Tungsten disulfide, $WS_2$ | 0.14 |
| Zinc iodide, $ZnI_2$ | 0.38 |

The basis for judging the performance of a potential lubricant material is the coefficient of friction. Potential lubricant materials are selected for further investigation if they exhibit a minimum friction coefficient of 0.22 or less during a preliminary screening run. This value corresponds to the average friction value for MLF-5 when it was evaluated under preliminary screening run conditions.

## IV. WEAR-LIFE INVESTIGATION

Wear-life investigation studies were started during the last quarter of this report period. The initial objectives of this work are to determine the effects of mating surface roughness and hardness on the friction and wear-life of MLF-5 and to accumulate "baseline" wear-life data on MLF-5 to serve as a basis for judging the performance of other solid lubricant films which are developed. Wear-life runs consist of sliding three lubricant-coated 440C stainless steel pellets over a 440C stainless steel wear-track plate.

The results of the wear-life runs completed to date are shown in Table IV.

TABLE IV

WEAR-LIFE OF MLF-5

Speed: 900 rpm (765 fpm)
Temperature: Room
Atmosphere: Air

| Run No. | Projected Area Load (psi) | Wear-Track Plate Hardness (Rockwell C) | Wear-Track Plate Roughness (rms) | Film Thickness (in.) | Wear-Life (hr.) | Wear-Life (hr/in x $10^4$) |
|---|---|---|---|---|---|---|
| 1 | 2.2 | 15 - 20 | 8 - 9 | 0.0005 | 110 | (22) |
| 2 | 2.2 | 15 - 20 | 2 - 3 | 0.0003 | 257 | (86) |
| 3 | 13.5 | 55 - 59 | 2 - 3 | 0.0004 | 26 | (6.5) |
| 4 | 13.5 | 55 - 59 | 6 - 7 | 0.0006 | 9.5 | (1.6) |
| 5 | 13.5 | 55 - 59 | 6 - 7 | 0.0005 | 9.7 | (1.9) |
| 6 | 13.5 | 55 - 59 | 6 - 7 | 0.0004 | 21.5 | (5.6) |

A wear-life pellet apparatus (see Fig. 2) was constructed for use in these investigations. In this apparatus a synchronous motor drives three pellets, rigidly mounted in a pellet holder, in sliding contact with a wear-track plate. The lubricant film to be investigated is applied to the flat ends of three cylindrically shaped pellets (1/4 in. in diameter). The pellet holder is driven through the O-ring belt and pulley arrangement at 900 rpm (765 fpm). The pellet holders and wear-track plates are identical with those used on the vacuum friction apparatus. During the first wear-life runs the apparatus was stopped, periodically, and film thickness was measured with a Magne-Gage. Film failure was deemed to occur when the thickness measurements indicated the film had worn through to the substrate.

The first two wear-life runs were performed with a load on the pellets of 50 gm/contact (2.2 psi) and different wear-track plate surface finishes (8 - 9 and 2 - 3 rms). The third run was performed with an increased load on the pellets or 300 gm/contact (13.5 psi) and a wear-track-plate surface finish of 2 - 3 rms. The last three runs were made with the 13.5 psi load and a wear-track surface finish of 6 - 7 rms. The results from these runs indicate that longer lubricant film wear-life is obtained with smoother mating surfaces and at the lighter load.

The curves (see Figs. 3 and 4) show that the wear characteristics of MLF-5 can be divided into three general phases. The first phase (run-in) lasts only a very short time (generally less than 20 min.) and is marked by rapid film wear. The second phase might be termed the useful wear-life of the film and is characterized by uniform (linear) wear. The third phase (film failure) is longer in duration than the "run-in" phase; however, it too is marked by rapid film wear.

Because some of the variation in wear-life (among the last three runs conducted at the same conditions) may have been caused by stopping for the film thickness measurements, the apparatus was then modified so that film failure could be sensed by a frictional torque measurement and thus avoid stopping for film thickness measurements. A thrust bearing was positioned between the wear-track plate and the apparatus frame, thereby, allowing the wear-track plate to "float" (see Fig. 3). The frictional torque reaction transmitted from the pellets to the wear-track plate is restrained by a calibrated spring. A cut-off switch is actuated to stop the run when a predetermined coefficient of friction is encountered. Experience has shown that a friction coefficient of approximately 0.3 is reached (for MLF-5) just prior to metal-to-metal contact. No runs have been completed at this time with the modified apparatus.

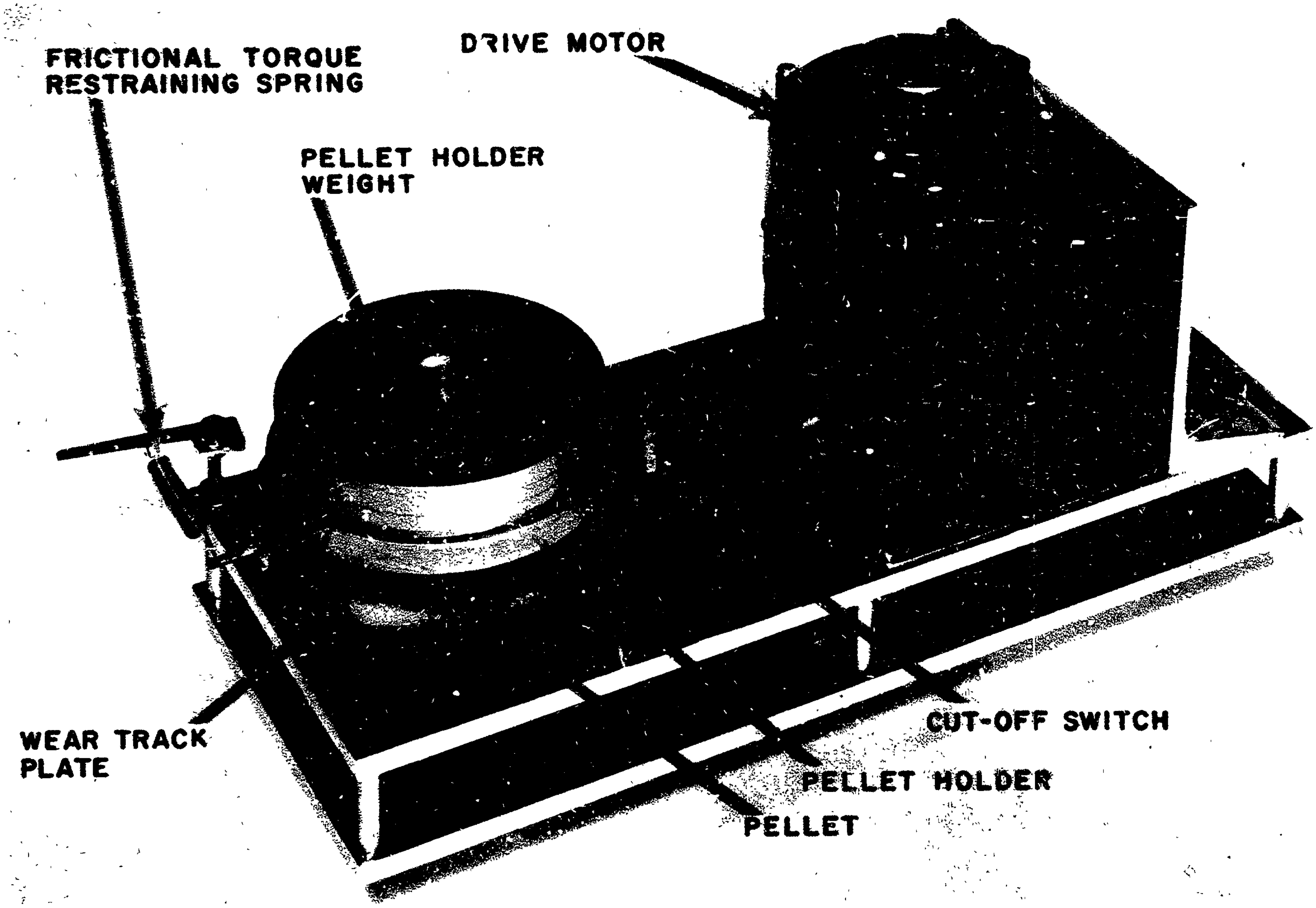



Fig. 2 - Wear-Life Pellet Apparatus

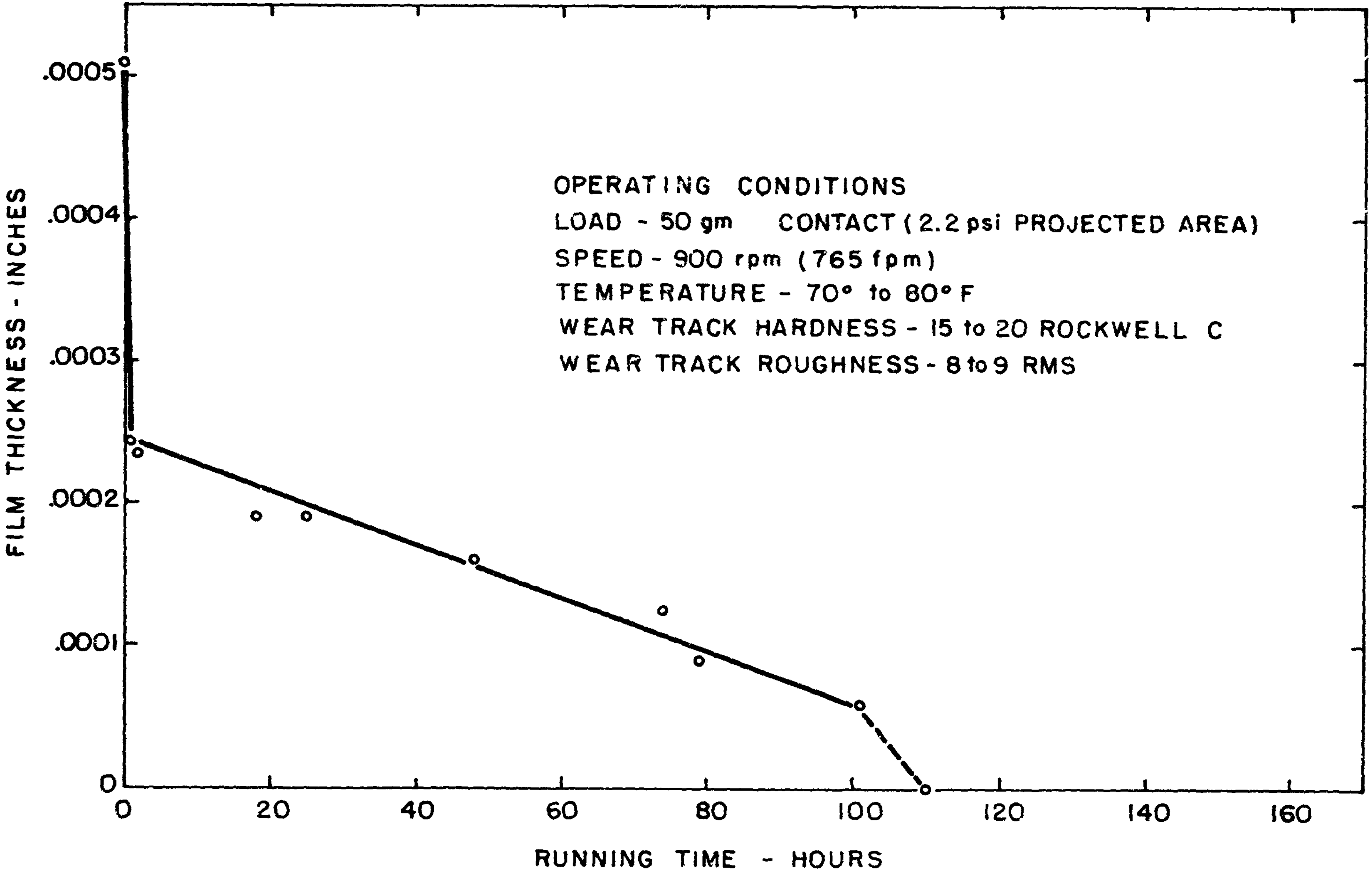



Fig. 3 - Wear Characteristic of MLF-5 Lubricant in Air (Run No. 1)

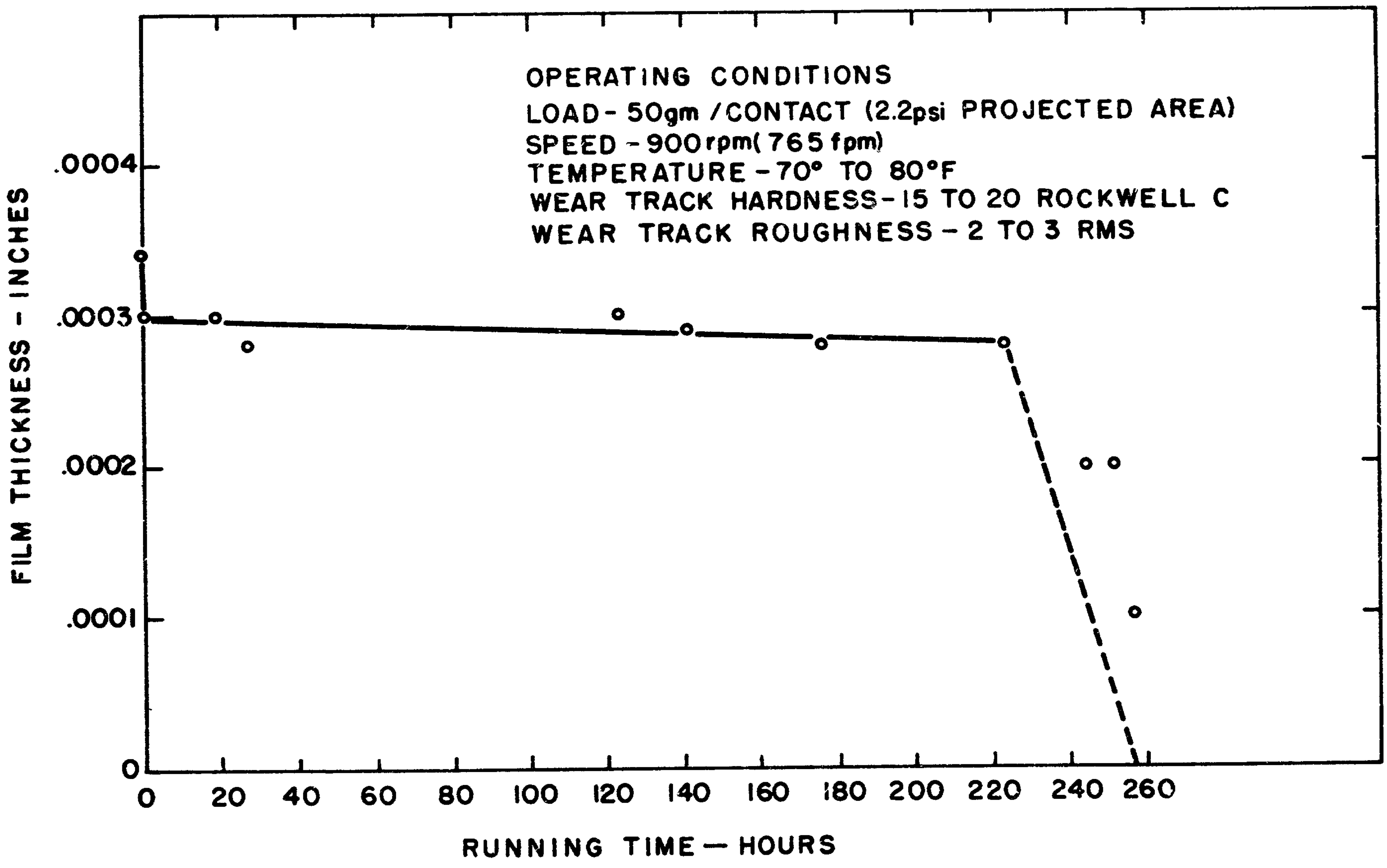



Fig. 4 - Wear Characteristic of MLF-5 Lubricant in Air Atmosphere
(Wear-Life Run No. 2)

Another wear-life device was designed and built to investigate the behavior of solid lubricant film for gear teeth applications. The apparatus is shown in Fig. 5. This device consists of a synchronous motor which drives a set of gears through an O-ring belt and pulley arrangement at speeds of 300, 600 or 900 rpm. The gears shown (32 pitch, 64 teeth, 2.06-in. O.D.) were coated with MLF-5 and run for approximately 500 hr. in an air atmosphere at no load. At the end of this run considerable film was left, but there were a few visible areas where the film was worn through to the substrate. It is planned to modify this apparatus so that wear-life runs may be conducted with the gears operating in a loaded condition.

## V. MODIFICATIONS OF THE VACUUM APPARATUS

The vacuum friction apparatus (see Fig. 6) which was designed, fabricated, and assembled during the first year of this program, was modified in several ways to improve its over-all performance.

The series-wound drive motor* was replaced with a special two-speed synchronous drive motor.** The installation of the replacement drive motor required minor modification of the drive frame and eliminated the need for motor speed control. The speed at which the series motor ran with a particular terminal voltage (adjusted with a variable transformer) was affected by the load on the motor. The friction between the pellets and wear-track plate is not constant during a run. Therefore, it was necessary to adjust almost continuously the variable transformer to keep the speed at the desired level (900 rpm). The motor was continually accelerating or de-accelerating and thus introducing errors in the frictional torque record.

Another modification was the replacement of the heater plate. The initial heater plate was made of aluminum and would lose its flatness after several thermal cycles from room temperature to 400°F. Since the actual surface area of the heater plate in contact with the wear-track plate decreased, the time required to raise the wear-track plate temperature from 80° - 400°F in vacuum increased. Therefore, a replacement heater was designed and fabricated to reduce this problem (see Fig. 7). The new heater consists of an electrical heater element (300 w.) bonded to the bottom of a spool-shaped piece of hot rolled steel with a sheet of silver. Two loops of 3/16 O.D. copper tubing are silver soldered in a groove in the outer edge of the spool to form a cooling coil.

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* Globe Universal, type GN, 6:1 gear head, output shaft speed 100 to 1,000 rpm, 115 v.

** Globe Special, 4:1 gear head, output shaft speed 300 rpm (6-pole) and 900 rpm (2-pole), 115 v.

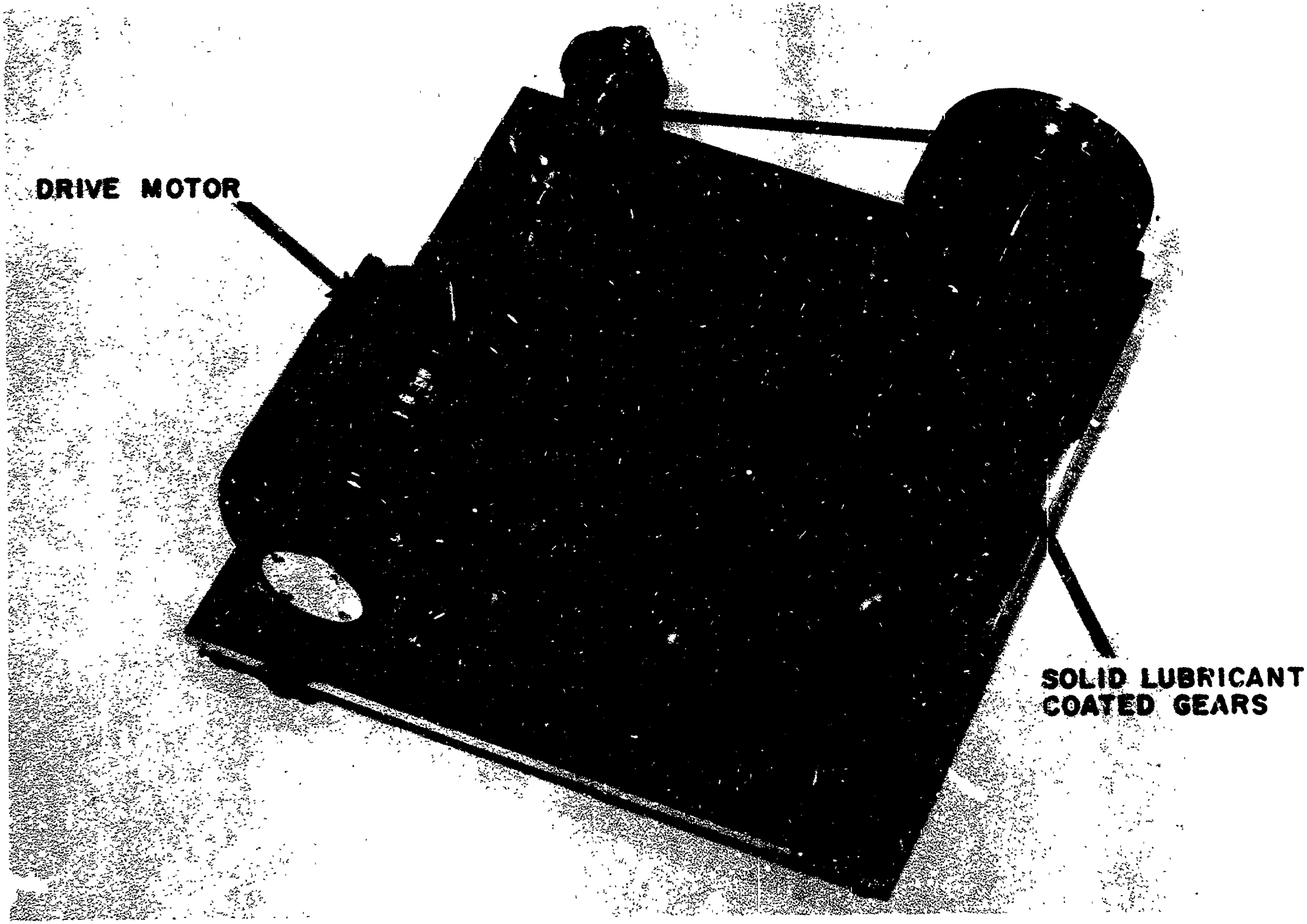


Fig. 5 - Wear-Life Gear Apparatus

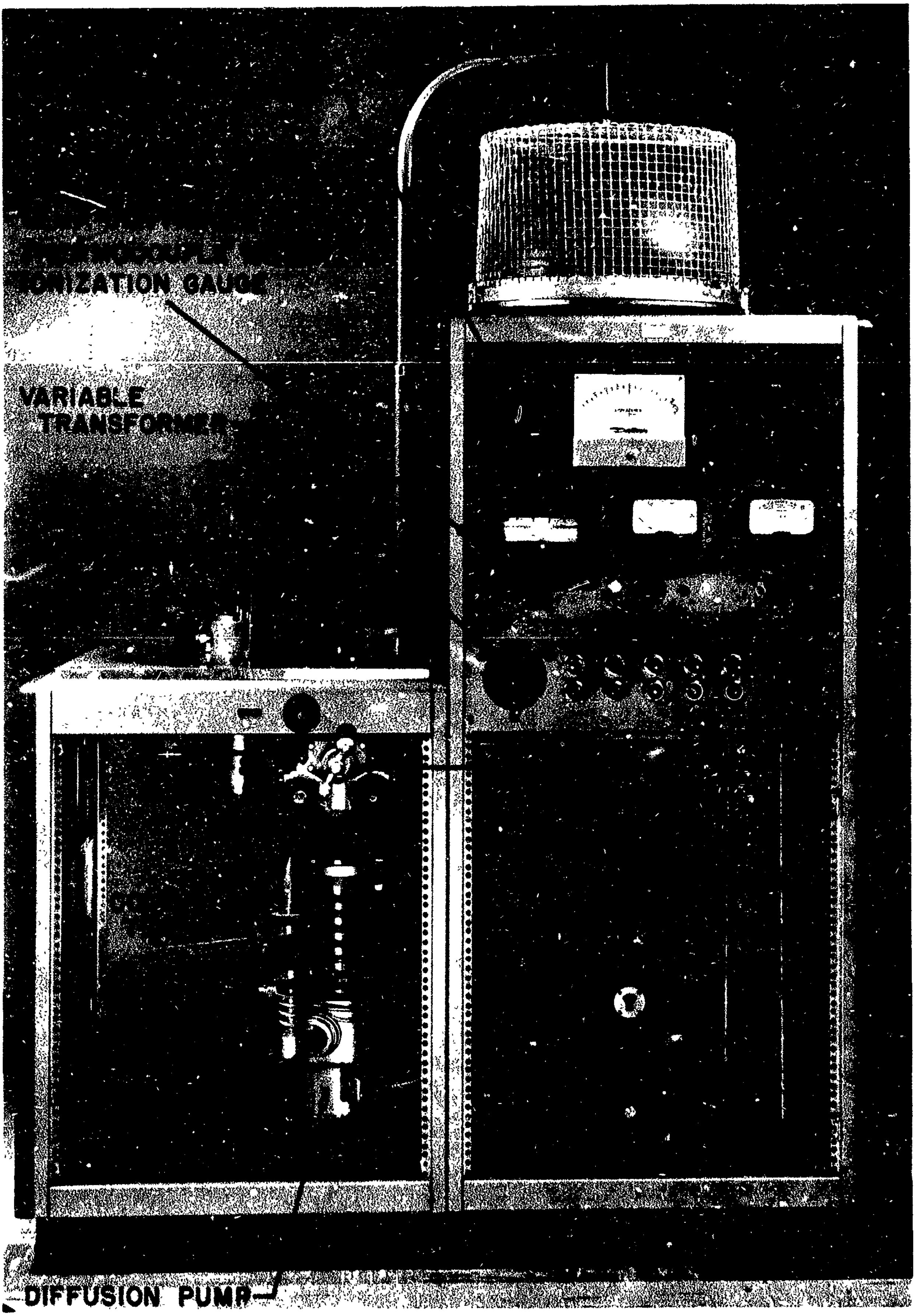


Fig. 6 - Vacuum Friction Apparatus

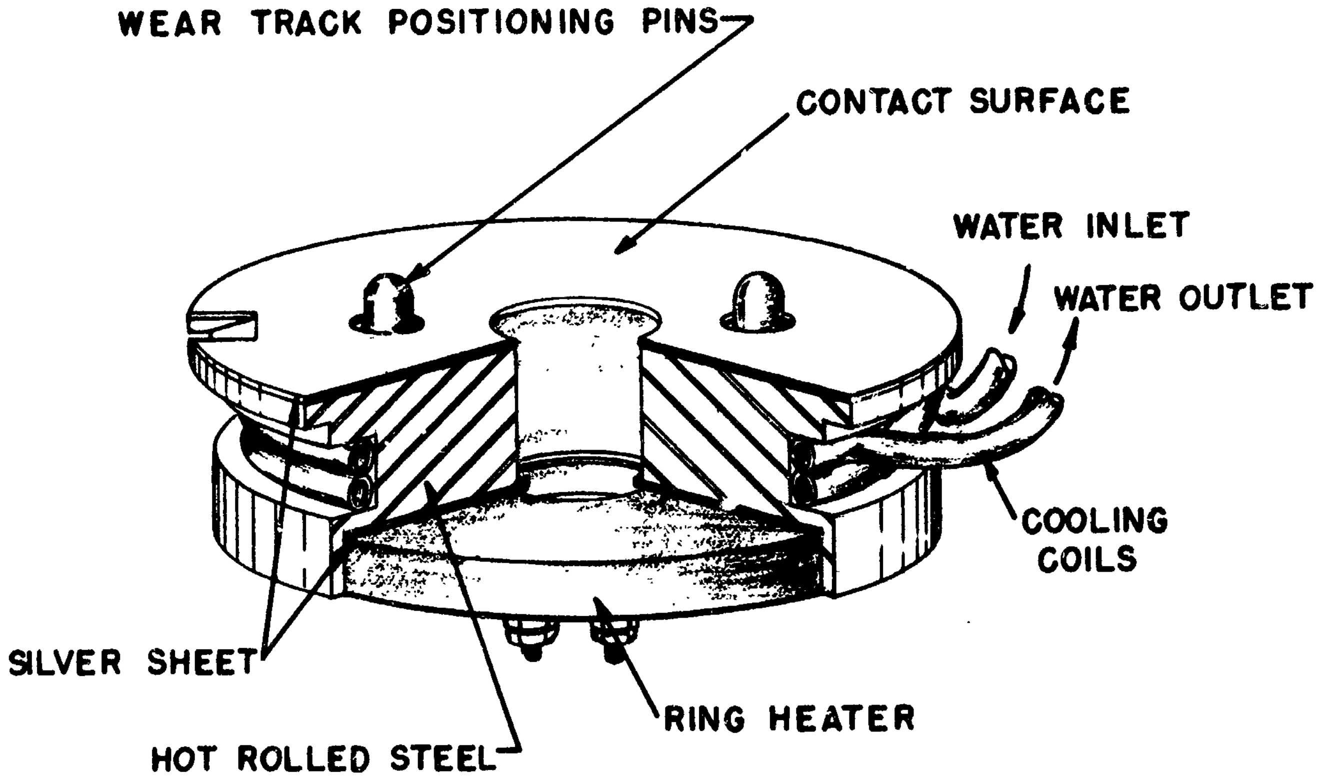


Fig. 7 - New Heater

A silver sheet is bonded to the top of the spool and two dowel pins are pressed in holes to position the wear track. The silver sheets were bonded to the heater element and the spool by heating to 750° - 1000°F while pressed together at approximately 1,000 psi. The silver surface that contacts the wear-track plate remains flat and allows the higher wear-track temperatures to be reached more quickly, particularly in vacuum. The new heater also permits the operating temperature to be extended to the subzero range. Liquid nitrogen can be introduced into the cooling coil to cool the wear-track plate. Friction and wear runs will be conducted at subzero temperatures only in a vacuum or a dry gaseous atmosphere.

All the pellet holders used on this program have been modified so that the pellets are rigidly held in position. In the initial pellet holder configuration the flat ends of the pellets were self-aligned with the wear-track plate. The self-aligning method of holding the pellets was not satisfactory for binder-material screening runs. The pellets chattered excessively during these runs because of the higher friction. They did not remain in contact with the wear-track plate. The rigidly held pellets worked well for both binder-screening runs and solid-lubricant friction and wear-life runs. The contact area between the pellets and wear-track surface is usually 85 - 100 per cent. In use, the pellets are all lapped flat and the solid lubricant film or binder is sprayed onto all three pellets, mounted in a pellet holder, at one time.

## VI. SPECIAL SOLID LUBRICANT FILM APPLICATION ACTIVITIES

During this report period, several miscellaneous parts were coated with bonded solid lubricant films for evaluation and use at NASA Marshall Space Flight Center and other concerns (see Table V). In nearly all cases, the solid lubricant must be sprayed to a certain thickness, and many times only on specific areas. Techniques for coating these parts with solid lubricant films had to be worked out for each different item. When over spray was not permitted, jigs and fixtures were made to shield surfaces which were not to be coated.

LOX compatibility tests were run on MLF-5 and another solid lubricant film which contained bismuth instead of gold at the Marshall Space Flight Center. The results indicated that both films were LOX compatible.

TABLE V

SOLID LUBRICANT COATED ITEMS

| Part | Quantity | Lubricant Film Applied | Destination |
|---|---|---|---|
| Test gimbal bearing | 18 | MLF-5* | MSFC |
| Test gimbal bearing | 2 | $MoS_2$, graphite, sodium silicate | MSFC |
| Test gimbal bearing | 1 | $MoS_2$, graphite, diamond, sodium silicate | MSFC |
| Test gimbal bearing | 4 | $MoS_2$, graphite, bismuth, sodium silicate | MSFC |
| Gimbal pin | 13 | MLF-5* | MSFC |
| Gimbal pin | 16 | MLF-5* | Pratt & Whitney Aircraft |
| Spider block | 4 | MLF-5* | Pratt & Whitney Aircraft |
| LOX impact disc | 30 | MLF-5* | MSFC |
| LOX impact disc | 30 | $MoS_2$, graphite, sodium silicate | MSFC |
| LOX impact disc | 30 | $MoS_2$, graphite, bismuth, sodium silicate | MSFC |
| Journal, 1/2 in. dia. | 4 | $MoS_2$, graphite, sodium silicate | MSFC |
| Valve stem | 6 | MLF-5* | MSFC |
| Ball bearing | 4 | MLF-5* | MSFC |
| Release clutch | 2 | MLF-5* | M.I.T., Lincoln Lab. |
| Pin puller | 4 | MLF-5* | M.I.T., Lincoln Lab. |
| Roller bearing | 1 | MLF-5* | M.R.I., Physics Div. |

* MLF-5, bonded inorganic solid lubricant film developed by MRI; see Appendix C, for description and application procedure.

## VII. FUTURE WORK

During the coming year we plan to:

1. Conduct the binder materials development studies with the potassium-silicate-base binder being modified by such additives as sodium borate, potassium phosphate, sodium fluoride, or aluminum phosphate.

2. Formulate and evaluate solid-lubricant films with current-lubricant materials ($MoS_2$, graphite, Au, etc.), and developed binder materials.

3. Formulate and evaluate solid lubricant films with potential-lubricant materials selected during this year's work and current-binder material (sodium silicate, K).

4. Establish wear-life "baseline" for MLF-5.

5. Study effects of mating surface roughness and hardness on friction and wear.

6. Search for potential lubricant materials and lubricant film components.

7. Develop and refine solid lubricant film application techniques.

8. Design, develop, and fabricate an ultrahigh vacuum apparatus capable of investigating the behavior of inorganic solid lubricants at environmental pressures below $10^{-10}$ Torr.

9. Investigate the behavior of inorganic solid film lubricants in the ultrahigh vacuum apparatus at pressures below $10^{-10}$ Torr.

10. Design a multistation vacuum apparatus for investigating the friction and wear behavior of developed solid-film lubricants applied to machine elements.

## BIBLIOGRAPHY


1. Hopkins, Vern, Gaddis, Donald, Kobett, Donald, and Hughes, Roland, "Research on Bearing Lubricants for Use in a High Vacuum," Annual Summary Report, 22 February 1961 to 22 March 1962.

2. Breed, L. S., and Morriss, F. V., "Research on High Metallo-organic Base Fluids," WADC TR 59-203, March 1959.

3. Lavik, Melvin T., "Ceramic Bonded Solid-Film Lubricants," WADD-TR-60-530, Parts I & II, January 1960, January 1961.

4. Lavik, Melvin T., "Physical and Chemical Properties of Ceramic Bonded Solid Lubricant Films," WADD-TR-60-530, Part III, February 1962.

# APPENDIX A

## FILM FAILURE DETECTION METHOD

Figure A-1 is a schematic of the circuit used to detect film failure in binder screening runs. The cured binder material electrically insulates the pellets rigidly mounted in the pellet holder from the wear track plate. Film failure is deemed to occur the instant metal-to-metal contact is made between the pellet holder and the wear track.

This instant is determined by upsetting a quiescent voltage charge on a condenser in series with a resistor and the pellet holder to common ground of the wear-track plate. An amplifier capacitively coupled to the resistor, amplifies any voltage variation such as may result from a momentary shorting of the specimen holder to wear plate. This voltage variation is then used to actuate a Schmitt trigger.

A Schmitt trigger consists of a two-stage regenerative coupled bistable switch, whose state of stability is dependent upon a quiescent stable input voltage, that when once disturbed will switch instantly to a new state of equilibrium.

This switching action drives a capacitively coupled thyratron into conduction energizing a Mercury relay in series with the plate of the thyratron. The closure of the relay contacts provide alarm conditions to end the test run by turning on an indicator lamp or shutting down the test.

After one test has been completed, a reset switch is operated to return the system to normal.

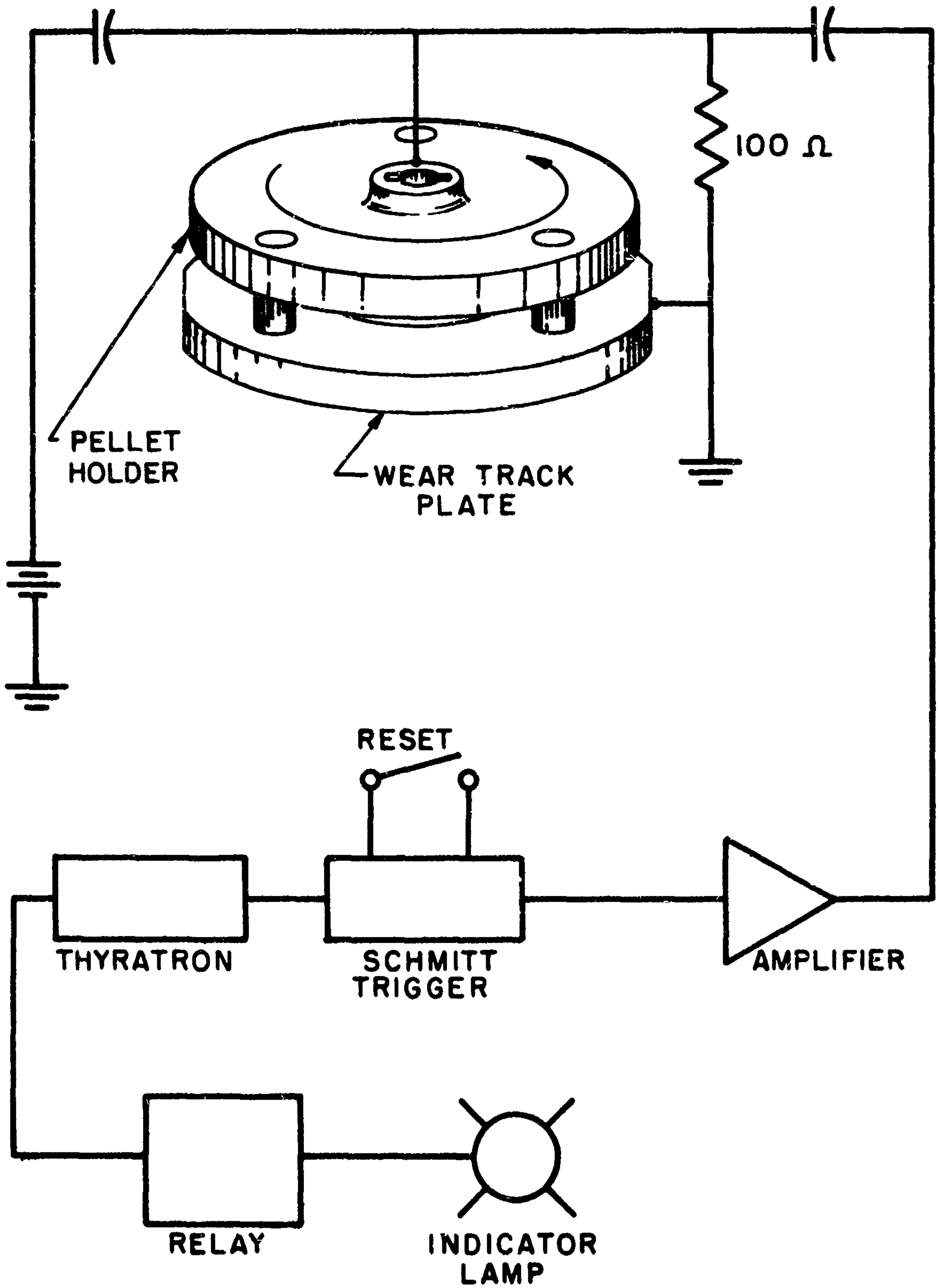


Fig. A-1 - Film Failure Detection Circuit Schematic

APPENDIX B

TABLES B-I - B-III

TABLE B-I

FRICTION AND WEAR-LIFE RESULTS FROM BINDER SCREENING RUNS IN AIR

Load: 50 gm/contact(2.2 psi) Speed: 900 rpm (765 fpm)
Wear-Track Temperature, Roughness, and Hardness: 70° to 90°F, 6 to 7 rms
55 to 59 Rockwell C (except as noted *)

| Binder | Additive-% by Wt. | Thickness (in.) | Friction Coefficient | Wear Life (min.) | Wear Life (min/in x$10^4$) |
|---|---|---|---|---|---|
| Sodium silicate, K | 0 | 0.00090 | 0.40-0.52 | 22 | (2.4) |
| Sodium silicate, K | 0 | 0.00085 | 0.25-0.37 | 18 | (2.1) |
| Sodium silicate, K | 0 | 0.00120 | 0.53-0.6+ | 23 | (1.9) |
| Sodium silicate, K | 0 | 0.00155 | 0.50-0.56 | 30 | (1.9) |
| Sodium silicate, K | 0 | 0.00080 | 0.41-0.51 | 6* | (0.7) |
| Sodium silicate, K | 0 | 0.00090 | 0.49-0.60 | 11* | (1.2) |
| Modified Kasil 88 | | | | | |
| Sample No.- 5 | $Na_3PO_4$- 5.6 | 0.00070 | 0.47-0.56 | 15 | (2.1) |
| - 5 | $Na_3PO_4$- 5.6 | 0.00100 | 0.46-0.56 | 10* | (1.0) |
| - 6 | $Na_3PO_4$- 8.2 | 0.00100 | 0.37-0.51 | 24 | (2.4) |
| - 6 | $Na_3PO_4$- 8.2 | 0.00100 | 0.51-0.6+ | 27* | (2.7) |
| - 7 | $Na_3PO_4$- 9.4 | 0.00110 | 0.51-0.59 | 13 | (1.2) |
| - 7 | $Na_3PO_4$- 9.4 | 0.00105 | 0.56-0.60 | 18* | (1.7) |
| - 8 | $Na_3PO_4$-10.6 | 0.00110 | 0.51-0.60 | 31 | (2.8) |
| - 8 | $Na_3PO_4$-10.6 | 0.00120 | 0.50-0.57 | 12* | (1.0) |
| - 9 | $Na_3PO_4$-11.8 | 0.00130 | 0.58-0.6+ | 15 | (1.1) |
| - 9 | $Na_3PO_4$-11.8 | 0.00110 | 0.55-0.6+ | 2* | (0.1) |
| -10 | $Na_3PO_4$-13.0 | 0.00100 | 0.45-0.50 | 6 | (0.6) |
| -10 | $Na_3PO_4$-13.0 | 0.00100 | 0.60-0.6+ | 4* | (0.4) |
| -11 | $Na_3PO_4$-14.1 | 0.00095 | 0.27-0.47 | 41 | (4.3) |
| -11 | $Na_3PO_4$-14.1 | 0.00095 | 0.37-0.51 | 22 | (2.3) |
| -11 | $Na_3PO_4$-14.1 | 0.00095 | 0.55-0.60 | 31 | (3.3) |
| -11 | $Na_3PO_4$-14.1 | 0.00125 | 0.55-0.6+ | 60 | (4.8) |
| -11 | $Na_3PO_4$-14.1 | 0.00095 | 0.55-0.6+ | 15* | (1.6) |
| -12 | $Na_3PO_4$-15.2 | 0.00110 | 0.41-0.50 | 20 | (1.8) |
| -12 | $Na_3PO_4$-15.2 | 0.00090 | 0.57-0.6+ | 27* | (3.0) |
| -13 | $Na_3PO_4$-16.2 | 0.00090 | 0.46-0.53 | 8 | (0.9) |
| -13 | $Na_3PO_4$-16.2 | 0.00080 | 0.46-0.6+ | 8 | (1.0) |
| -14 | $Na_3PO_4$-17.3 | 0.00130 | 0.48-0.51 | 9 | (0.7) |
| -14 | $Na_3PO_4$-17.3 | 0.00100 | 0.57-0.6+ | 59 | (5.9) |
| -14 | $Na_3PO_4$-17.3 | 0.00080 | 0.60-0.6+ | 20 | (2.5) |
| -14 | $Na_3PO_4$-17.3 | 0.00080 | 0.60-0.6+ | 30 | (3.7) |

TABLE B-I (Continued)

| Linder | Additive-% by Wt. | Thickness (in.) | Friction Coefficient | Wear Life (min.) | Wear Life (min/in x$10^4$) |
|---|---|---|---|---|---|
| Modified Kasil 88 | | | | | |
| Sample No.-15 | $Na_3PO_4$-18.3 | 0.00110 | 0.50-0.54 | 15 | (1.4) |
| -15 | $Na_3PO_4$-18.3 | 0.00112 | 0.48-0.6+ | 17 | (1.5) |
| -15 | $Na_3PO_4$-18.3 | 0.00090 | 0.28-0.51 | 58 | (6.4) |
| -15 | $Na_3PO_4$-18.3 | 0.00110 | 0.50-0.6+ | 40 | (3.6) |
| -16 | $Na_3PO_4$-19.2 | 0.00100 | 0.58-0.60 | 3 | (0.3) |
| -16 | $Na_3PO_4$-19.2 | 0.00120 | 0.30-0.55 | 20 | (1.7) |
| -17 | $Na_3PO_4$-20.2 | 0.00100 | 0.41-0.6+ | 12 | (1.2) |
| -17 | $Na_3PO_4$-20.2 | 0.00130 | 0.60-0.6+ | 15 | (1.1) |
| -18 | $Na_3PO_4$-21.2 | 0.00130 | 0.51-0.57 | 3 | (0.2) |
| -18 | $Na_3PO_4$-21.2 | 0.00140 | 0.44-0.56 | 11 | (0.8) |
| -19 | $Na_3PO_4$-22.0 | 0.00095 | 0.45-0.59 | 13 | (1.4) |
| -19 | $Na_3PO_4$-22.0 | 0.00097 | 0.55-0.58 | 6 | (0.6) |
| -20 | $Na_3PO_4$-22.9 | 0.00115 | 0.43-0.55 | 11 | (0.9) |
| -20 | $Na_3PO_4$-22.9 | 0.00137 | 0.44-0.50 | 5 | (0.4) |
| -21 | $K_3PO_4$ - 3.3 | 0.00125 | 0.45-0.50 | 8 | (0.6) |
| -21 | $K_3PO_4$ - 3.3 | 0.00110 | 0.55-0.6+ | 12 | (1.1) |
| -23 | $K_3PO_4$ - 9.4 | 0.00110 | 0.45-0.6+ | 16 | (1.4) |
| -23 | $K_3PO_4$ - 9.4 | 0.00107 | 0.50-0.60 | 19 | (1.8) |
| -25 | $K_3PO_4$ -14.7 | 0.00113 | 0.48-0.54 | 11 | (0.9) |
| -25 | $K_3PO_4$ -14.7 | 0.00155 | 0.48-0.6+ | 26 | (1.7) |
| -27 | $K_3PO_4$ -19.4 | 0.00112 | 0.45-0.54 | 15 | (1.3) |
| -27 | K3PO4 -19.4 | 0.00100 | 0.52-0.6+ | 10 | (1.0) |
| -29 | K3PO4 -23.7 | 0.00119 | 0.32-0.52 | 15 | (1.3) |
| -29 | K3PO4 -23.7 | 0.00110 | 0.39-0.48 | 24 | (2.2) |
| -31 | $K_3PO_4$ -27.5 | 0.00130 | 0.52-0.55 | 14 | (1.1) |
| -31 | $K_3PO_4$ -27.5 | 0.00130 | 0.55-0.6+ | 10 | (0.8) |
| -33 | $K_3PO_4$ -31.0 | 0.00130 | 0.38-0.51 | 10 | (0.8) |
| -33 | K3PO4 -31.0 | 0.00155 | 0.52-0.55 | 23 | (1.5) |
| -35 | $K_3PO_4$ -34.1 | 0.00155 | 0.55-0.6+ | 11 | (0.7) |
| -37 | $K_3PO_4$ -37.0 | 0.00155 | 0.52-0.6+ | 33 | (2.1) |
| -37 | $K_3PO_4$ -37.0 | 0.00155 | 0.32-0.50 | 12 | (0.8) |
| -40 | NaF - 0 | 0.00112 | 0.60-0.6+ | 19 | (1.7) |
| -40 | NaF - 0 | 0.00110 | 0.60-0.6+ | 13 | (1.2) |
| -41 | NaF - 6.5 | 0.00112 | 0.60-0.6+ | 19 | (1.7) |
| -41 | NaF - 6.5 | 0.00112 | 0.60-0.6+ | 5 | (0.4) |
| -42 | NaF -12.1 | 0.00115 | 0.50-0.6+ | 4 | (0.3) |
| -42 | NaF -12.1 | 0.00108 | 0.45-0.50 | 7 | (0.6) |
| -43 | NaF -17.1 | 0.00095 | 0.60-0.6+ | 3 | (0.3) |
| -43 | NaF -17.1 | 0.00093 | 0.52-0.55 | 2 | (0.2) |

TABLE B-I (Concluded)

| Binder | Additive- % by Wt. | Thickness (in.) | Friction Coefficient | Wear Life (min.) | Wear Life (min/in x$10^4$) |
|---|---|---|---|---|---|
| Modified Kasil 88 | | | | | |
| Sample No.-44 | NaF -21.6 | 0.00112 | 0.54-0.6+ | 11 | (1.0) |
| -44 | NaF -21.6 | 0.00110 | 0.52-0.6+ | 14 | (1.3) |
| -45 | NaF -25.6 | 0.00098 | 0.60-0.6+ | 4 | (0.4) |
| -45 | NaF -25.6 | 0.00112 | 0.55-0.6+ | 31 | (2.8) |
| -46 | NaF -29.2 | 0.00115 | 0.55-0.6+ | 15 | (1.3) |
| -46 | NaF -29.2 | 0.00128 | 0.55-0.6+ | 82 | (6.4) |
| -47 | NaF -32.6 | 0.00128 | 0.55-0.6+ | 27 | (2.1) |
| -47 | NaF -32.6 | 0.00112 | 0.55-0.60 | 1 | (1.0) |
| -48 | NaF -35.5 | 0.00092 | 0.60-0.6+ | 23 | (2.5) |
| -48 | NaF -35.5 | 0.00112 | 0.50-0.6+ | 18 | (1.6) |
| -49 | NaF -38.2 | 0.00110 | 0.32-0.52 | 6 | (0.5) |
| -50 | NaF -40.8 | 0.00098 | 0.55-0.6+ | 48 | (4.9) |
| -50 | NaF -40.8 | 0.00110 | 0.45-0.50 | 5 | (0.4) |
| -56 | $Na_2B_4O_7$- 8.2 | 0.00130 | - | 1 | (0.8) |
| -56 | $Na_2B_4O_7$- 8.2 | 0.00155 | 0.45-0.54 | 10 | (0.6) |
| -57 | $Na_2B_4O_7$- 6.3 | 0.00112 | 0.60-0.6+ | 14 | (1.2) |
| -57 | $Na_2B_4O_7$- 6.3 | 0.00084 | 0.6[illegible].6+ | 12 | (1.4) |
| -58 | $Na_2B_4O_7$- 4.3 | 0.00090 | 0.4[illegible]0.50 | 6 | (0.7) |
| -58 | $Na_2B_4O_7$- 4.3 | 0.00090 | 0.50-0.6+ | 7 | (0.8) |
| -59 | $Na_2B_4O_7$- 2.2 | 0.00080 | 0.60-0.6+ | [illegible] | (0.7) |
| -59 | $Na_2B_4O_7$- 2.2 | 0.00100 | 0.60-0.6+ | 1 | (0.1) |
| -60 | $Na_2B_4O_7$- 1.1 | 0.00085 | 0.54-0.60 | 3 | (0.3) |
| -60 | $Na_2B_4O_7$- 1.1 | 0.00105 | 0.54-0.55 | 8 | (0.8) |

* Denotes wear track hardness of 15 to 20 Rockwell C.

TABLE B-II

WEAR-LIFE RESULTS FROM SPECIAL BINDER RUNS

Wear-Track Hardness and Roughness - 55 to 59 Rockwell C and 4 to 6 rms
Binder - Potassium Silicate (Kasil 88) + 11.8% by weight $Na_3PO_4$

| Binder Application Method | Thickness (in.) | Environmental* Atmosphere | Wear Life (min.) | Wear Life (min/in x $10^4$) |
|---|---|---|---|---|
| A | 0.00110 | Air | 12 | (1.1) |
| A | 0.00105 | " | 41 | (3.9) |
| A | 0.00110 | " | 14 | (1.3) |
| B | 0.00108 | " | 42 | (3.9) |
| B | 0.00120 | " | 4 | (0.3) |
| C | 0.00069 | " | 15 | (2.2) |
| C | 0.00061 | " | 4 | (0.6) |
| C | 0.00062 | " | 11 | (1.8) |
| C | 0.00077 | " | 25 | (3.3) |
| D | 0.00132 | " | 43 | (3.3) |
| D | 0.00116 | " | 20 | (1.7) |
| D | 0.00112 | " | 24 | (2.1) |
| D | 0.00110 | " | 48 | (4.4) |
| D | 0.00107 | " | 30 | (2.8) |
| D | 0.00094 | " | 7 | (0.7) |
| E | 0.00095 | " | 15 | (1.6) |
| E | 0.00112 | " | 34 | (3.0) |
| F | 0.00110 | " | 35 | (3.2) |
| F | 0.00095 | " | 7 | (0.7) |
| E | 0.00103 | $N_2$ | 62 | (6.0) |
| E | 0.00112 | " | 102 | (9.1) |
| E | 0.00112 | " | 106 | (9.5) |
| E | 0.00110 | " | 60 | (5.5) |
| F | 0.00110 | " | 88 | (8.0) |
| F | 0.00140 | " | 135 | (9.6) |
| F | 0.00120 | " | 39 | (3.2) |
| F | 0.00127 | " | 46 | (3.6) |

* Normal atmospheric pressure.

TABLE B-III

FRICTION AND WEAR-LIFE RESULTS FROM BINDER SCREENING RUNS IN NITROGEN

| Binder | Additive-% by Wt. | Thickness (in.) | Friction Coefficient | Wear Life (min.) | Wear Life (min/in x $10^4$) |
|---|---|---|---|---|---|
| Kasil 88 | - 0 | 0.00113 | 0.37-0.6+ | 38 | (3.4) |
| " " | - 0 | 0.00128 | 0.35-0.6+ | 35 | (2.7) |
| " " | - 0 | 0.00110 | 0.40-0.55 | 34 | (3.0) |
| Modified Kasil 88 | | | | | |
| Sample No. 3 | $Na_3PO_4$- 2.9 | 0.00105 | 0.34-0.46 | 27 | (2.6) |
| " " 3 | " - 2.9 | 0.00120 | 0.35-0.6+ | 27 | (2.2) |
| " " 5 | " - 5.6 | 0.00120 | 0.20-0.6+ | 58 | (4.8) |
| " " 5 | " - 5.6 | 0.00120 | 0.30-0.6+ | 71 | (5.9) |
| " " 6 | " - 8.2 | 0.00130 | 0.25-0.6+ | 92 | (7.1) |
| " " 6 | " - 8.2 | 0.00110 | 0.50-0.6+ | 68 | (6.2) |
| " " 7 | " - 9.4 | 0.00115 | 0.24-0.6+ | 60 | (5.2) |
| " " 7 | " - 9.4 | 0.00095 | 0.33-0.6+ | 94 | (9.8) |
| " " 7 | " - 9.4 | 0.00100 | 0.20-0.55 | 60 | (6.0) |
| " " 8 | " -10.6 | 0.00112 | 0.39-0.6+ | 53 | (4.7) |
| " " 8 | " -10.6 | 0.00090 | 0.55-0.6+ | 34 | (3.8) |
| " " 8 | " -10.6 | 0.00100 | 0.44-0.6+ | 11 | (1.1) |
| " " 10 | " -13.0 | 0.00110 | 0.27-0.6+ | 43 | (2.9) |
| " " 10 | " -13.0 | 0.00110 | 0.31-0.6+ | 19 | (1.7) |
| " " 10 | " -13.0 | 0.00110 | 0.30-0.6+ | 49 | (4.5) |
| " " 11 | " -15.2 | 0.00110 | 0.23-0.40 | 5 | (0.4) |
| " " 11 | " -15.2 | 0.00110 | 0.25-0.50 | 68 | (6.2) |
| " " 11 | " -15.2 | 0.00125 | 0.35-0.6+ | 77 | (6.2) |
| " " 11 | " -15.2 | 0.00085 | 0.33-0.6+ | 58 | (6.8) |
| " " 11 | " -14.1 | 0.00094 | 0.28-0.6+ | 9 | (0.9) |
| " " 11 | " -14.1 | 0.00140 | 0.30-0.6+ | 96 | (6.9) |
| " " 11 | " -15.2 | 0.00100 | 0.30-0.6+ | 35 | (3.5) |
| " " 12 | " -15.2 | 0.00120 | 0.36-0.50 | 52 | (4.3) |
| " " 12 | " -15.2 | 0.00120 | 0.18-0.6+ | 40 | (3.3) |
| " " 13 | " -16.2 | 0.00110 | 0.50-0.6+ | 45 | (4.1) |
| " " 13 | " -16.2 | 0.00100 | 0.36-0.6+ | 39 | (3.9) |
| " " 14 | " -17.3 | 0.00105 | 0.23-0.6+ | 48 | (4.6) |
| " " 14 | " -17.3 | 0.00100 | 0.25-0.6+ | 23 | (2.3) |
| " " 14 | " -17.3 | 0.00085 | 0.32-0.55 | 36 | (4.2) |
| " " 15 | " -18.3 | 0.00110 | 0.45-0.6+ | 16 | (1.5) |
| " " 15 | " -18.3 | 0.00100 | 0.55-0.6+ | 50 | (5.0) |
| " " 15 | " -18.3 | 0.00110 | 0.32-0.6+ | 71 | (6.5) |
| " " 15 | " -18.3 | 0.00125 | 0.6+ | 50 | (4.0) |
| " " 16 | " -19.2 | 0.00100 | 0.40-0.6+ | 23 | (2.3) |
| " " 16 | " -19.2 | 0.00120 | 0.40-0.6+ | 38 | (3.2) |

TABLE B-III (Concluded)

| Binder | Additive - % by Wt. | Thickness (in.) | Friction Coefficient | Wear Life (min.) | Wear Life (min/in x $10^4$) |
|---|---|---|---|---|---|
| Modified Kasil 88 | | | | | |
| Sample No. 21 | $K_3PO_4$- 3.3 | 0.00100 | 0.32-0.6+ | 23 | (2.3) |
| " " 21 | " - 3.3 | 0.00130 | 0.24-0.6+ | 33 | (2.5) |
| " " 21 | " - 3.3 | 0.00120 | 0.55-0.6+ | 4 | (0.3) |
| " " 21 | " - 3.3 | 0.00125 | 0.55-0.6+ | 1 | (0.1) |
| " " 23 | " - 9.4 | 0.00125 | 0.55-0.6+ | 8 | (0.6) |
| " " 23 | " - 9.4 | 0.00130 | 0.55-0.6+ | 5 | (0.4) |
| " " 23 | " - 9.4 | 0.00120 | 0.29-0.6+ | 24 | (2.0) |
| " " 23 | " - 9.4 | 0.00095 | 0.6+ | 5 | (0.5) |
| " " 23 | " - 9.4 | 0.00120 | 0.25-0.55 | 19 | (1.6) |
| " " 25 | " -14.7 | 0.00130 | 0.18-0.6+ | 57 | (4.4) |
| " " 25 | " -14.7 | 0.00110 | 0.30-0.6+ | 28 | (2.5) |
| " " 25 | " -14.7 | 0.00095 | 0.37-0.6+ | 3 | (0.3) |
| " " 25 | " -14.7 | 0.00100 | 0.34-0.41 | 8 | (0.8) |
| " " 26 | " -17.1 | 0.00125 | 0.20-0.6+ | 94 | (7.5) |
| " " 27 | " -19.4 | 0.00120 | 0.27-0.6+ | 25 | (2.3) |
| " " 27 | " -19.4 | 0.00115 | 0.34-0.6+ | 88 | (7.6) |
| " " 29 | " -23.7 | 0.00140 | 0.36-0.6+ | 20 | (1.4) |
| " " 29 | " -23.7 | 0.00120 | 0.30-0.6+ | 26 | (2.2) |
| " " 31 | " -27.5 | 0.00120 | 0.6+ | 7 | (0.6) |
| " " 31 | " -27.5 | 0.00100 | 0.33-0.6+ | 11 | (1.1) |
| " " 33 | " -31.0 | 0.00130 | 0.36-0.6+ | 7 | (0.5) |
| " " 33 | " -31.0 | 0.00140 | 0.42-0.6+ | 14 | (1.0) |
| " " 56 | $Na_2B_4O_7$-8.2 | 0.00130 | 0.27-0.6+ | 17 | (1.3) |
| " " 56 | " -8.2 | 0.00110 | 0.28-0.45 | 14 | (1.3) |
| " " 57 | " -6.3 | 0.00090 | 0.19-0.6+ | 27 | (3.0) |
| " " 58 | " -4.3 | 0.00100 | 0.30-0.6+ | 42 | (4.2) |

# APPENDIX C

## MLF-5 LUBRICANT APPLICATION PROCEDURE

## Materials

The following materials are used in MLF-5 solid film lubricant:

1. Molybdenum disulphide powder: This product is available from Alpha Molykote Corporation, Stamford, Connecticut, under the name "Molykote, Type Z, Mil-M-7866A (ASG) Lubricant." This product has an average particle size of approximately 7 microns and must be ordered from the supplier specially selected for low oil contamination.

2. Graphite powder: This product is available from the Joseph Dixon Crucible Company, Jersey City 3, New Jersey, under the name "Dixon 635 Flake Graphite."

3. Gold powder: This product is available from Fisher Scientific Company, New York 14, New York, under the name "Precipitated Gold, G-50."

4. Sodium silicate: This product is available from Philadelphia Quartz Company, Philadelphia, Pennsylvania, under the name, "Product K, %$Na_2O$: %$SiO_2$ ratio 1:2.9, Be 47°." This product has a definite shelf life and shall be used within the date specified on the container.

5. Distilled water: Distilled water is used as a fluid dispersant.

## Lubricant and Substrate Preparation

All solid materials are passed through a clean No. 400 mesh screen individually. Only the solid materials so screened are used in MLF-5 lubricant. The materials are then weighed individually in a clean suitable container according to the relative weights as shown in Schedule I.

### SCHEDULE I - COMPOSITION OF MLF-5 BY WEIGHT

| | | |
|---|---|---|
| 1. | Molybdenum disulphide | 10 parts |
| 2. | Graphite | 1 part |
| 3. | Gold | 5 parts |
| 4. | Sodium silicate | 7 parts |
| 5. | Distilled water | 60 parts |

The weighed materials are combined in a clean container adding distilled water last. The lubricant mixture is thoroughly stirred a minimum of 1 hr. to properly wet the solids in powder form. Continuous stirring is required until the lubricant has been applied to the substrate.

Parts to be coated with MLF-5 lubricant are machine-finished to a surface roughness of 8 to 16 rms. The surfaces so machined are then liquid-honed to a roughness of 8 to 13 rms.

Substrate cleaning prior to lubricant application follows the sequence shown in Schedule II. The parts to be cleaned are handled in a manner that bare skin and other contaminating surfaces do not come in contact with the surfaces to be coated.

SCHEDULE II - SURFACE CLEANING SEQUENCE

| Step | Environment |
|---|---|
| 1. Wash | Boiling detergent solution |
| 2. Rinse | Distilled water |
| 3. Dry | Air |
| 4. Rinse (3 consecutive baths) | Acetone, Reagent Grade |
| 5. Dry | Air |
| 6. Store until ready for coating | Methanol, Reagent Grade or, Benzene, Reagent Grade |

## Application Process

The MLF-5 lubricant is applied to the substrate using a suitable spray gun or air brush. The reservoir of the spray device is stirred continuously. Pressure is supplied to the spray device from a dry nitrogen source. The nozzle of the spray device is adjusted to deliver a very fine mist. The rate of application must be such that the lubricant appears to dry on contact, and that moist spots do not appear on the coated surface. Drying is aided by directing an infrared heat lamp upon the coated surface. However, the coated surface does not exceed a temperature of 140°F during application.

In general, a coat or layer of lubricant approximately 0.0001 to 0.0004 in. thick may be applied in one continuous spray application without damaging moisture accumulating on the substrate surface. Several coats or layers may be applied until a desired thickness is achieved.

The optimum total thickness of the film of lubricant depends upon the particular application and/or available clearances. Small instrument ball bearings may allow clearance for no more than 0.0004 to 0.0006 in., while larger, heavily loaded bearings may allow as much as 0.002 in. total film thickness. Film thickness may be controlled by frequent measurements with a magnetic thickness gage during the lubricant application process. (A suitable instrument is available from the American Instrument Company, Bethesda, Maryland, under the name "Simco-Brenner Magne-Gage.")

Once the film of lubricant has been applied to the substrate the curing cycle commences. The time-temperature sequence shown in Schedule III is followed to properly cure the lubricant film.

SCHEDULE III - CURING TEMPERATURES IN AIR ATMOSPHERE

| Step | Temperature | Time |
|---|---|---|
| 1 | 65° to 100°F | 1 hr. |
| 2 | 180°F | 4 hr. |
| 3 | 300°F | 8 to 16 hr. |
| 4 | Cool down from 300° to 150°F | 1 hr. minimum |

The parts are removed from the furnace and stored in a dry, oil-free atmosphere. The parts are handled so that the lubricated surfaces do not come in contact with bare skin or other contaminating surfaces.